

## **FS 2400. Wastewater Sampling**

See also the following Standard Operating Procedures:

- FA 1000 Regulatory Scope and Administrative Procedures for Use of FDEP SOPs
- FC 1000 Cleaning/Decontamination Procedures
- FD 1000-9000 Documentation Procedures
- FM 1000 Field Planning and Mobilization
- FQ 1000 Field Quality Control Requirements
- FS 1000 General Sampling Procedures
- FS 2000 General Aqueous Sampling

### **1. INTRODUCTION AND SCOPE**

Use this Standard Operating Procedure (SOP) during field investigations to ensure that representative wastewater samples are collected.

All persons conducting wastewater sampling must be knowledgeable of the protocols in this SOP. The sampling organization must maintain documentation demonstrating that all sampling personnel have been adequately trained, have copies of this SOP and are familiar with the sampling protocols.

Prior to mobilizing, the sampler must decide what kind of samples to collect, what analytes are to be analyzed, and where to collect the samples. The sampler must take care to ensure that the sampling location is correct and that the samples are representative of the discharge. The sample site locations and sample types must be consistent with the permit(s), if applicable, or as described in a work plan.

General guidance for the operation, calibration and maintenance of autosamplers is included in this SOP. The individual user should consult the equipment's operating and maintenance manuals for operating instructions, maintenance and troubleshooting of the equipment.

### **2. General Cautions**

2.1. In order to obtain a representative sample, collect samples where wastewater flow is adequately mixed. Flow mixing is particularly important for ensuring uniformity. In general, use these criteria to evaluate the location:

2.2. Sample in the center of the flow where velocity is highest and there is little possibility of solids settling.

2.2.1. Collect the sample at a depth between 40% and 60% of the total depth where the turbulence is maximized. Avoid the water surface or the channel bottom.

2.3. Take precautions when collecting samples near a weir to avoid solids that tend to collect upstream and floating oil and grease that accumulate downstream.

2.4. If the sample is not to be tested for volatile organics or will not be affected by stripping of dissolved gases, you may use mechanical stirring or introduce a stream of air into the waste stream.

2.5. In sampling from wide conduits, consider cross-sectional sampling. Use a dye as an aid in determining the most representative sampling point(s). Note: Experienced personnel should be consulted for the type of dye and appropriate protocols.

2.6. If manual compositing is employed, mix the contents of the individual sample bottles thoroughly before pouring the individual aliquots into the final composite container.

2.7. If the sample is taken from an effluent tap, allow the tap to run for one to two minutes to allow the settled solids to flush from the line. Reduce the flow to 500 mL/min before collecting the samples.

2.8. Sampling and flow measuring are integrally related. The sampler must know the wastewater flow variability before a sampling program can be initiated. Whether to use a flow proportional or time composite sampling scheme depends on the variability of the wastewater flow. If a sampler knows or suspects significant variability in the wastewater flow or knows nothing about the facility, a flow proportional sample should be collected; otherwise, a time composite sample would be acceptable.

2.9. Prior to sampling, examine the flow measuring system (primary flow device, totalizer, recorder). See section 18 on Flow Measurement in the "EPA Environmental Investigations Standard Operating Procedures and Quality Assurance Manual," May 1996 with 1997 revisions. If the flow measuring system is unacceptable, the sampler may have to install a flow-measuring device. If the flow measuring system is acceptable, samples can be collected by the appropriate method.

2.10. Using dedicated equipment at each sampling station will avoid cross-contamination between sampling stations. If using sampling equipment for multiple sampling locations, and volatile organics, extractable organics or metals are not analytes of concern, decontaminate the sampling device between sites by rinsing with ample amounts of site water. See FC 1131 for decontamination protocols for the sampling equipment once it is returned to the base of operations or if metals or organics are analytes of concern.

## **FS 2410. EQUIPMENT AND SUPPLIES**

Select sampling equipment based on the parameters of interest, the specific equipment use and the available equipment. Select sampling equipment from table FS 2400-1. Sample containers and sampling equipment must be of the appropriate construction material as described in FS 1000, Tables FS 1000-1 and FS 1000-2.

Clean all sampling equipment and obtain clean sample containers using the appropriate protocols specified in FC 1000.

1. **MANUAL SAMPLING:** Manual sampling is used for collecting grab samples and/or for immediate in-situ field analyses. However, it can be used in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to evaluate unusual waste stream conditions.

### **2. AUTOMATIC SAMPLERS**

2.1. A wide variety of automatic samplers are commercially available. Most have the following five interrelated subsystem components:

2.1.1. Sample Intake Subsystem: The sample intake gathers representative samples from the sampling stream.

2.1.1.1. The intake is usually the end of a plastic suction tube that should also be resistant to physical damage from large objects in the flow stream. Non-leaching Tygon tubing is most often used.

2.1.1.2. Use Teflon tubing if collecting samples for organics (including all extractable organics and pesticides). Fix the tubing end to a piece of conduit or a pole bent to hold the sample port in the waste stream at the correct location to get a representative sample. Support the tubing so either the supporting pole or the method of attachment does not contaminate the incoming sample. If a base is employed to hold the end of the tubing in the waste stream it must be constructed of non-contaminating materials and any screening material must still allow the collection of representative samples.

2.1.2. Sample Gathering Subsystem: Automatic samplers provide one of three basic gathering methods:

2.1.2.1. Mechanical: Mechanical gathering subsystems are usually built into place and include devices such as cups on cables, calibrated scoops, and paddle wheels with cups. Although these systems obstruct the stream flow, they take into account site-specific considerations, such as high sampling lift and wide/deep channel flow. Because of the mechanical system employed, these units require periodic maintenance.

2.1.2.2. Forced Flow: Forced flow gathering subsystems are often built into place as permanent sampling facilities. Like the mechanical gathering subsystems, they may obstruct the stream flow. They also require periodic inspection and maintenance. However, forced flow subsystems have the advantage of being able to sample at great depths. In addition, because this gathering system uses air pressure to transport the sample, it may be ideal for sample collection in potentially explosive environments.

2.1.2.3. Suction Lift: The suction lift is the most widely used type of sample gathering subsystem due to its versatility and minimal effect on flow patterns. Suction lifts are limited to 25 vertical feet or less because of internal friction losses and atmospheric pressure. Collect at least 100 mL of sample each time the pump is actuated.

2.1.3. Sample Transport Subsystem: The sample is usually transported from the sample intake to the collection bottle by a plastic tube called the "sample transport subsystem." Make sure the sampler is placed well above the effluent stream to ensure the tubing runs in a taut, straight line to prevent pooling of liquid. Take care to avoid sharp bends and twists in the transport line.

2.1.4. Sample Storage Subsystem: The sample storage subsystem can accommodate either a single large collection bottle or a number of smaller collection bottles. Samplers with individual bottles for discrete collection are usually equipped with a cassette that rotates to fill the bottle during sampling. The total sample volume storage capability should be at least 2 gallons (7.6 liters). Some samplers have a capacity of up to 5 gallons.

2.1.4.1. If using an unrefrigerated sampler unit and the project calls for cooling during collection, storage subsystems must be large enough to provide space for sufficient ice to chill the sample during the entire collection event.

2.1.5. Controls and Power Subsystem: The control units allow for the selection of time or flow compositing, or a continuous sampling method. The automatic samplers most widely used have encapsulated solid-state controls. This configuration minimizes the effects of unfavorable environments that may be encountered in the field, such as high humidity and corrosiveness. These units are also sealed so they may be used with minimal risk in potentially explosive environments. In addition, sealed units protect the controls if the sampler is accidentally submerged.

2.2. Automatic sampling equipment must meet the following requirements:

2.2.1. Properly clean sampling equipment to avoid cross- contamination that could result from prior use (see FC 1140 for specific cleaning procedures).

2.2.2. If samples for extractable organics are to be collected, all parts of the sampler that come in contact with the wastewater stream must conform to the construction material requirements in FS 1000, Tables FS 1000-1 and FS 1000-2.

2.2.3. If the preservation requirements for a particular component specify that a sample be thermally preserved, cool the sampler so samples are kept at 4°C during the sampling period. Use ice or refrigeration units in the sampler.

2.2.4. The sampler must be able to collect a large enough sample for all analyses needed. Additionally, split samples may also be necessary.

2.2.5. If using a peristaltic pump, collect a minimum of 100 mL each time the sampler is activated.

2.2.6. The sampler should provide a lift up to at least 20 feet and the sampler should be adjustable so that volume is not a function of the pumping head.

2.2.7. The pumping velocity must be adequate to transport solids and not allow solids to settle.

2.2.8. The automatic sampler must provide for line purging after each sample is drawn to prevent contamination of subsequent samples. The minimum intake line inside diameter must be at least 1/4 inch, which is large enough to lessen chances of clogging but small enough to maintain velocity and to avoid solids settling.

2.2.9. The tubing of the sample transport system should be at least 1/4 inch inside diameter to maintain adequate flow and to prevent plugging. Tubing should be sized so that a velocity of at least two feet per second can be maintained. The sample line must be automatically purged after each sample is collected.

2.2.10. Ensure that an adequate power supply is available to operate the sampler during the entire sampling event.

2.2.11. Sample collection vessels, either large composite or discrete sample containers, must be constructed of materials appropriate for the tests to be performed.

2.3. In addition to the above requirements, consider the following when selecting automatic sampling equipment:

2.3.1. Convenience of Installation and Maintenance: Always carefully handle sampling equipment and maintain equipment in accordance with the manufacturer's instructions. Careless handling and poor maintenance cause most equipment failures.

2.3.2. Equipment Security: Security is important when sampling is done as part of an enforcement proceeding. Manhole locations where battery operated equipment may be installed and the cover replaced will aid in maintaining security. If sampling equipment must be left unattended, the sampler should be provided with a lock or seal that, if broken or disturbed, would indicate that tampering had occurred and sample integrity possibly compromised.

2.3.3. Operation in Cold or Hot Weather

2.3.3.1. Cold Weather: Intake lines may freeze when collecting samples during extremely cold days. If necessary, use heat tape or place the sampler inside a thermostatically controlled, electrically heated enclosure. In the absence of any special heating equipment, wrap the sampler with eight to nine inches of insulation and place in a manhole or wet well to prevent freezing.

2.3.3.2. Hot Weather: During hot weather, choose a shaded or cooled place for the sampler, if possible. Wrapping the sampler with insulation may also help. Painting the sampler white will reflect some heat. If using refrigerated or iced units, refill the automatic sampler with ice or check to see that the refrigeration unit is operating before leaving the site.

## **FS 2420. SAMPLE TYPES**

There are two primary types of samples: 1) grab samples; and 2) composite samples. Each type has distinct advantages and disadvantages. While grab sampling allows observation of unusual conditions that may exist during discharge, this method is labor intensive and impractical when sampling is performed at many locations over extended periods of time. When sampling a large number of locations, the use of automatic samplers is more practical.

Automatic samplers also help reduce human error, specifically in complex sampling activities such as flow proportional sampling, and reduce exposure to potentially hazardous environments. The primary disadvantage to automatic sampling is the cost of the equipment and maintenance requirements (samplers tend to fail, battery quits, no refrigeration, etc.).

In order to obtain a more complete characterization of a specific facility's effluent, the two sample types can be used independently or in combination. The sampler must weigh advantages and disadvantages when choosing between the use of grab or composite sampling methods if the sample type has not already been specified in a permit or project work plan.

## **FS 2421. Grab Samples**

1. This is an individual sample collected over a period of time, usually all in one motion, generally not exceeding 15 minutes. The 15-minute time limit applies to aqueous samples only. No time limit applies to the collection of solid samples (e.g., residuals). Grab samples may be used to determine consistency among an industry's self-monitoring data and to corroborate the results of composite samples.

2. Grab samples represent the conditions that exist at the moment the sample is collected and do not necessarily represent conditions at any other time. Grab sampling is the preferred method of sampling under the following conditions:

2.1. A snapshot of the wastewater quality at a particular instant in time is desired.

- 2.2. The water or wastewater stream is not continuous (e.g., batch discharges or intermittent flow).
- 2.3. The characteristics of the water or waste stream are known to be constant or nearly so.
- 2.4. When the waste conditions are relatively constant over the period of discharge. In lieu of complex sampling activities, a grab sample provides a simple and accurate method of establishing waste characteristics.
- 2.5. The sample is to be analyzed for analytes whose characteristics are likely to change significantly with time (e.g., dissolved gases, microbiological tests, pH, etc.).
- 2.6. The sample is to be collected for analytes such as Oil and Grease, bacteriological tests or other parameters listed in number 3 of this section where the compositing process could significantly affect the actual concentration.
- 2.7. Data on maximum/minimum concentrations are desired for a continuous water or wastewater stream.
- 2.8. When identifying and tracking slug loads and spills.

3. If required, measure the following parameters on grab samples or in-situ. NOTE: If the permit specifies a composite sample for any of the parameters mentioned below, **FOLLOW THE PERMIT CONDITIONS.**

Cyanide	Oil and Grease
Residual Chlorine	pH
Dissolved constituents in field-filtered samples (ortho-phosphorus, metals, etc.)	Specific Conductance
Dissolved Oxygen and other dissolved gases	Un-ionized Ammonia
Microbiological Parameters	Volatile Organic Compounds
TRPHs	Temperature

### **FS 2422. Composite Samples**

1. A composite sample is a sample collected over time, formed either by continuous sampling or by mixing discrete samples. Composite samples reflect the average characteristics during the compositing period.
2. Composite samples are used when stipulated in a permit or when:
  - 2.1. The water or wastewater stream is continuous;
  - 2.2. Analytical capabilities are limited;
  - 2.3. Determining average pollutant concentration during the compositing period;
  - 2.4. Calculating mass/unit time loadings; or
  - 2.5. Associating average flow data to parameter concentrations.
3. Composite samples may be collected individually at equal time intervals if the flow rate of the sample stream does not vary more than plus or minus ten percent of the average flow

rate, or they may be collected proportional to the flow rate. The permit or work plan may specify which composite sample type to use, either time composites or flow proportional composites. The compositing methods, all of which depend on either continuous or periodic sampling, are described in the following discussions.

3.1. Time Composite Sample: Time composite samples are based on a constant time interval between samples. A time composite sample can be collected manually or with an automatic sampler. This type of composite is composed of discrete sample aliquots collected in one container at constant time intervals. This method provides representative samples when the flow of the sampled wastewater stream is constant. This type of sample is similar to a sequential composite sample described in number 3.3 of this section.

3.2. Flow Proportional Composite Sample: Flow proportional samples can be collected automatically with an automatic sampler and a compatible pacing flow measuring device, semi-automatically with a flow chart and an automatic sampler capable of collecting discrete samples, or manually. There are two methods used to collect this type of sample:

3.2.1. Method 1: Collect a constant sample volume per stream flow (e.g., a 200 mL sample collected for every 5,000 gallons of stream flow) at time intervals proportional to stream flow. This method provides representative samples of all waste streams when the flow is measured accurately.

3.2.2. Method 2: Collect a sample by increasing the volume of each aliquot as the flow increases, while maintaining a constant time interval between the aliquots (e.g., hourly samples are taken with the sample volume being proportional to the flow at the time the sample is taken).

3.3. Sequential Composite Sample: Sequential composite samples are composed of discrete samples taken into individual containers at constant time intervals or constant discharge increments. For example, samples collected every 15 minutes are composited for each hour.

3.3.1. The 24-hour composite is made up from the individual one-hour composites. Each of the 24 individual samples is manually flow-proportioned according to the flow recorded for the hour that the sample represents. Each flow-proportioned sample is then added to the composite samples. The actual compositing of the samples is done by hand and may be done in the field or the laboratory. In most cases, compositing in the field is preferable since only one sample container must be cooled, and then transported to, and handled, in the laboratory. A 24-hour composite is frequently used since an automatic sampler can easily collect the individual samples.

3.3.2. A variation of the 24-hour composite is to collect a constant volume of sample taken at constant discharge increments, which are measured with a totalizer. For example, one aliquot is collected for every 10,000 gallons of flow.

3.3.3. Sequential sampling is useful to characterize the waste stream because you can determine the variability of the wastewater constituents over a daily period. For example, for pretreatment studies you can visually determine when high strength wastes are being discharged from a facility or when heavy solid loads are being discharged during a 24-hour cycle. You can measure the pH throughout the day. The value of this type of sampling must be weighed against the manpower constraints and sampling goals.

3.4. Continuous Composite Sample: Collected continuously from the waste stream. The sample may be a constant volume that is similar to the time composite, or the volume may vary in proportion to the flow rate of the waste stream, in which case the sample is similar to the flow proportional composite.

3.5. Areal Composite: A sample composited from individual grab samples collected on an areal or cross-sectional basis. Areal composites must be made up of equal volumes of grab samples; each grab sample must be collected in an identical manner. Examples include residual samples from grid system points on a land application site, water samples collected at various depths at the same point or from quarter points in a stream, etc.

## **FS 2430. WASTEWATER SAMPLING TECHNIQUES**

The following protocols must be used when collecting wastewater samples. All general protocols applicable to aqueous sampling detailed in FS 2000 must be adhered to when following the wastewater sampling procedures addressed below.

1. **MANUAL SAMPLING**: Use manual sampling for collecting grab samples for immediate in-situ field analyses. Also use manual sampling in lieu of automatic equipment over extended periods of time for composite sampling, especially when it is necessary to observe and/or note unusual waste stream conditions.

1.1. Surface Grab Samples: DO NOT use sample containers containing premeasured amounts of preservatives to collect surface grab samples. If the sample matrix is homogeneous, grab samples are an effective and simple technique. If homogeneity is not apparent based on flow or vertical variations (and should never be assumed) then consider other sample types to characterize the waste stream.

Where practical, use the actual sample container submitted to the laboratory for collecting samples to be analyzed for oil and grease, volatile organic compounds (VOCs) and bacteriological samples. This procedure eliminates the possibility of contaminating the sample with an intermediate collection container.

The use of unpreserved sample containers as direct grab samplers is encouraged because the same container can be submitted for laboratory analysis after appropriate preservation. This procedure reduces sample handling and eliminates potential contamination from other sources (e.g., additional sampling equipment, environment).

### **1.1.1. Grab Directly Into Sample Container**

1.1.1.1. Submerge the container, neck first, into the water.

1.1.1.2. Invert the bottle so the neck is upright and pointing into the water flow (if applicable).

1.1.1.3. Return the filled container quickly to the surface.

1.1.1.4. Pour out a few mL of sample downstream of the sample collection point. This procedure allows for addition of preservatives and sample expansion. This step must not be followed for volatile organics or other analytes where headspace is not allowed in the sample container.

1.1.1.5. Add preservatives, securely cap container, and label.

1.1.2. If sample containers are attached to a pole via a clamp, submerge the sample container and follow steps 1.1.1.3 – 1.1.1.5 as outlined in this section omitting steps 1.1.1.1 and 1.1.1.2.

1.1.3. Sampling with an Intermediate Vessel or Container: If the sample cannot be collected directly into the sample container to be submitted to the laboratory or if the laboratory provides prepreserved sample containers, use an unpreserved sample container or an intermediate vessel to obtain the sample. Intermediate vessels may be precleaned beakers, buckets or dippers. These vessels must be constructed appropriately including any poles or extension arms used to access the sample location.

1.1.3.1. Collect sample as outlined in steps 1.1.1.1 – 1.1.1.5 of this section.

1.1.3.2. Pole mounted containers of appropriate construction are used to sample at distances away from shore, boat, etc. or at different depths. Follow the protocols in 1.1.2 of this section for collecting samples.

1.1.3.3. Make sure the intermediate vessel gets rinsed where appropriate.

1.1.4. Peristaltic Pump and Tubing: This technique is not acceptable for Oil & Grease, TRPHs or Volatile Organic Compounds. Extractable Organics can be collected through the pump if flexible interior-wall Teflon, polyethylene or PP tubing is used in the pump head or if used with the organic trap setup as shown in FS 2200, Figure FS 2200-1.

1.1.4.1. Lower appropriately precleaned tubing to a depth 6 – 12 inches below water surface, where possible.

1.1.4.2. Pump 3 – 5 tube volumes through the system to acclimate the tubing.

1.1.4.3. Fill the individual sample bottles via the discharge tubing being careful not to remove the inlet tubing from the water.

1.2. Mid-depth Grab Samples: Mid-depth samples or samples taken at a specific depth can approximate the conditions throughout the entire water column. The equipment that may be used for this type of sampling consists of depth specific sampling devices (Kemmerer, Niskin, Van Dorn, etc.); pumps with tubing; or double-check valve bailers. When purchasing and choosing a device for a particular sampling event, please be aware that certain construction material details may preclude its use for certain analytes (see Tables FS 1000-1 and FS 1000-2).

**NOTE THAT ALL RELATED COMPONENTS (STOPPERS, ETC.) MUST BE CONSTRUCTED OF INERT MATERIAL AS WELL IF VOLATILE OR EXTRACTABLE ORGANICS ARE TO BE SAMPLED.**

1.2.1. Kemmerer, Niskin, and Van Dorn Type Devices

1.2.1.1. Many Kemmerer samplers are constructed of plastic and rubber that preclude their use for all volatile and extractable organic sampling. Some of the newer devices are constructed of stainless steel or are all Teflon or Teflon-coated. These would be acceptable for all analyte groups without restriction.

1.2.1.2. Before lowering the sampler, measure the water column to determine maximum depth and sampling depth.

1.2.1.3. Mark the line attached to the sampler with depth increments so that the sampling depth can be accurately recorded.

1.2.1.4. When dropping the sampler to the appropriate depth, do it slowly so that sediments are not stirred up.

1.2.1.5. Once the desired depth is reached send the messenger weight down to trip the mechanism.

1.2.1.6. Retrieve the sampler slowly.

1.2.1.7. Fill the individual sample bottles via the discharge tube. Sample bottles must be handled as described in sections 1.1.1.4 - 1.1.1.5 of this section.

1.2.2. Double Check-Valve Bailer: Collect samples using double check-valve bailers if the data requirements do not necessitate a sample from a strictly discrete interval of the water column. Bailers with an upper and lower check-valve can be lowered through the water column and water will be continually displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. This technique may not be successful in strong currents.

1.2.2.1. Follow the same protocols outlined in section 1.2.1 of this section except that a messenger weight is not applicable.

1.2.2.2. Although not designed specifically for this kind of sampling, it will be acceptable when a mid-depth sample is required.

1.2.2.3. Note: this sampler does not perform as well as the devices described above or the pump and tubing described in the next section.

1.2.2.4. As the bailer is dropped through the water column, water will be displaced through the body of the bailer. The degree of displacement is dependent upon the check-valve ball getting out of the way and allowing water to flow freely through the bailer body.

1.2.2.5. Drop the bailer slowly to the appropriate depth. Upon retrieval, the (two) check valves seat, preventing water from escaping out of, or entering, the bailer.

1.2.2.6. Fill the individual sample bottles via the discharge tube. Handle sample bottles as described in 1.1.1.4 - 1.1.1.5 of this section.

1.2.3. Peristaltic Pump and Tubing: If necessary, collect samples using a pump, either power or hand operated, to withdraw a sample from the water or wastewater stream. The most portable pump for this technique is a (12 volt) peristaltic pump. Appropriately precleaned silastic tubing is required in the pump head and polyethylene, Tygon, etc. tubing is attached to the pump.

This technique is not acceptable for Oil & Grease, TRPHs or Volatile Organic Compounds. Extractable Organics can be collected through the pump if flexible interior-wall Teflon, polyethylene or PP tubing is used in the pump head or if used with the organic trap setup as shown in FS 2200, Figure FS 2200-1.

1.2.3.1. Measure the water column to determine the maximum depth and the sampling depth.

1.2.3.2. Tie the tubing to a stiff pole or weight it down so the tubing placement will be secure. Do not use a lead weight. Any dense, non-contaminating, non-interfering material will work (brick, stainless steel weight, etc.). Tie the weight with a lanyard (braided or monofilament nylon, etc.) so that it is located below the inlet of the tubing.

1.2.3.3. Turn the pump on and allow several tubing volumes of water to be discharged before taking the first sample.

1.2.3.4. Sample bottles must be handled as described in 1.1.1.4 - 1.1.1.5 of this section.

2. AUTOMATIC SAMPLERS: Use automatic samplers when several sites are to be sampled at frequent intervals or when a continuous sample is required. Composite samplers can be used to collect time composite or flow proportional samples. In the flow proportional mode, some samplers are activated by a compatible flow meter. Refer to your specific flow meter operating manual for details on meter operation. For older models, flow proportional samples can be collected using a discrete sampler and a flow recorder and manually compositing the individual aliquots in flow proportional amounts.

### 2.1. Installing and Programming the Composite Sampler

2.1.1. Use all new or precleaned pump tubing each time the sampler is brought to the field and set up. Select tubing construction for the pump head and sampling train according to the analytes of interest and the allowable construction materials specified in FS 1000, Tables FS 1000-1 and FS 1000-2.

2.1.1.1. Cut the proper length of precleaned Teflon or Tygon tubing.

2.1.1.2. Collect equipment blanks at a frequency of 5% of the samples by passing analyte-free water through the equipment that is exposed to the sample. Collect a minimum of one blank each year.

2.1.1.3. Put the collection sieve and tubing in the appropriate sample location in the wastewater stream, using conduit if necessary to hold it in place. Ensure the supporting conduit does not contaminate the incoming sample water.

2.1.1.4. Program the sampler per manufacturer's directions and as required in the permit or work plan conditions.

2.1.1.5. For a time composite sample, program the sampler to collect a minimum of 100 mL for each sample interval. Adjust the volume collected according to the duration of the sampling event, the sampling interval and the size of the container.

2.1.1.6. For a flow proportional sample, program the sampler to collect a minimum of 100 mL for each sample interval, with the interval predetermined based on the flow of the waste stream.

2.1.1.7. Automatic Sampler Security: A lock or seal may be placed on the sampler to prevent or detect tampering. However, this procedure does not prevent tampering with the sampler tubing. See additional discussions on sample security in FS 2410, section 2.3.

### 2.2. Sample Acquisition

2.2.1. At the end of each sampling period, stir the contents of the compositing jug (sample) and siphon contents (poured if no visible solids) into the respective containers. If the sampler was set up to collect discrete samples ensure that the contents of each container are adequately mixed while pouring the sample into the sample container.

2.2.2. Immediately preserve the sample, if required, cap and label the sample container.

2.3. Long-Term Deployment of Automatic Composite Samplers: In certain sampling situations, automatic composite samplers are permanently installed at the sample stations and remain in the field for months or even years. Under these conditions, there are specific sampling issues that need to be addressed.

2.3.1. Sample Preservation

2.3.1.1. If the only analyte of interest is Total Phosphorus, and the project is unrelated to an NPDES permit, the sample must be chemically preserved with  $\text{H}_2\text{SO}_4$  but it need not be cooled to 4°C with wet ice.

2.3.1.2. The acid must be in the container prior to drawing the first composite sample into the container. When using large (i.e., 3 gallon) composite sample containers, and there is potential for the sample size to vary greatly due to variable flow rates at the site, the volume of acid for preservation should be small (e.g., 1 to 2 mLs of 50%  $\text{H}_2\text{SO}_4$ ). **Do not over acidify the sample.** Upon sample pick-up, if needed, add additional acid to achieve the proper pH adjustment for preservation.

2.3.1.3. If parameters other than Total Phosphorus are to be analyzed, appropriate additional preservation (e.g., cooling with ice or refrigeration) is required.

2.3.2. Cleaning Requirements

2.3.2.1. Clean composite sampler containers after collection of each composite sample using cleaning solutions and procedures specified in FC 1140, sections 5 through 8.

2.3.2.2. Composite sample containers may be cleaned either in the field or in a fixed based operation. Demonstrate cleaning effectiveness by collecting equipment blanks on the composite sample containers according to the frequency specified in FQ 1000. Collect sampler container equipment blanks by adding a minimum of 1 L of analyte-free water to the cleaned sample container, mix the water thoroughly within the container and then pour off an aliquot for analysis.

2.3.2.3. Replace tubing at a minimum of every six months. Inspect the tubing each time the composite sample-container is picked up. If there is evidence of loss of elasticity or discoloration or other conditions that would impact the quality of the sample (such as algal growth), then replace the tubing prior to the end of the six-month interval. Collect an equipment blank each time new tubing is installed in the auto sampler. Collect this equipment blank by passing analyte free water through the entire length of the new tubing being deployed at each station. If the tubing is being replaced for multiple autosamplers at the same time, one equipment blank may be collected on the entire length of replacement tubing. Collect this equipment blank by passing analyte-free water through the entire length of new tubing.

## **FS 2440.      BIOSOLIDS**

These procedures are to be followed when sampling sludges generated by domestic wastewater treatment processes. These procedures are also applicable to other industrial treatment processes where the characteristics or the nature of the solids generated may vary considerably. Typically sludges will be flowing through pipes, moving on conveyor belts or stored in piles or bins.

The number of samples to be collected, the frequency of monitoring, and the type of sample

(grab or composite) will be established by the appropriate FDEP rule, the permit or project work plan. Biosolid samples collected for metals must be composite samples and samples for pathogens and percent volatile solids must be grabs to comply with Chapter 62-640, F.A.C. Collect composite samples in such a manner that the sample represents, as close as possible, the quality of the biosolids that will be disposed of or used. Seven grab samples must be collected for fecal coliform analyses to meet Class B Alternative 1 requirements.

**1. GENERAL CONSIDERATIONS**

1.1. Be aware of the characteristics of the sludge stream to allow the collection of representative samples. The solids content and viscosity of the waste stream will dictate the method by which the most representative samples will be obtained.

1.2. Obtain well mixed samples that are representative of the entire flow.

**2. SAFETY PRECAUTIONS:** Follow safety precautions as outlined in "Standard Methods for the Examination of Water and Wastewater," 18<sup>th</sup> Edition, Section 1060A. Individuals performing sampling must be trained in the microbiological hazards of domestic wastewater and sludge and in safety precautions to take when sampling.

2.1. Wear gloves when handling or sampling sewage sludges.

2.2. Clean the sample containers, gloves and your hands before delivering the samples to others.

2.3. Take precautions not to touch other areas of your body while sampling.

2.4. It is recommended that all personnel handling domestic sludges have their inoculations up to date.

2.5. Take extreme care when sampling enclosed areas for biosolids due to the potential for the buildup of dangerous gases.

**FS 2441. Equipment and Supplies**

See the introduction to FS 2410 and Table FS 2400-1 for the selection of equipment and supplies.

1. Sample containers used to collect samples must be widemouth bottles that can be capped or flexible plastic containers.

2. Sewage sludges can generate gases so do not completely fill the container.

2.1. Do not fill the sample bottles for the oxygen uptake test more than half full to provide oxygen for respiration of the organisms in the sludge.

2.2. Samples to be analyzed for anaerobic vector attraction reduction must not be exposed to oxygen more than momentarily. The containers must be completely full and have closures that can pop off or containers that are made of flexible plastic.

2.3. Fill sample bottles for oxygen uptake rate only half full to provide oxygen for the respiration of the sludge.

**FS 2442. Sampling Procedures**

1. **FREE-FLOWING SLUDGE:** Sewage sludges below 7% solids behave as moderately viscous liquids. They are heterogeneous and settle upon standing but the settling is slow and is overcome by adequate mixing. These types of sludges may be flowing in pipes, in a processing tank such as a clarifier or digester, stored in tanks or in lagoons. It is preferable to

sample liquid sewage sludges as they are being transferred from one vessel to another, preferably sampled downstream of a pump that serves to mix the sludge thoroughly.

1.1. Sampling from Taps

1.1.1. The selection of a tap for a sampling point is adequate if:

1.1.1.1. The tap is located on the side of the main discharge pipe.

1.1.1.2. The sampling point is over 10 pipe diameters downstream from the pipe inlet or the tap is downstream from a pump.

1.1.1.3. The flow is turbulent.

1.1.1.4. The tap is large enough to ensure that an adequate quantity of sample is pulled from the cross-section of the flow.

1.1.2. Thoroughly flush the sample port before collecting the sample.

1.1.3. Draw the sample fast enough to minimize the amount of thinned out fluid from the pipe wall.

1.1.4. Transfer the liquid sludge samples to a widemouth bottle or flexible plastic sample container.

1.1.5. Pails, buckets, pitchers or cylinders may be used as intermediate sample collection devices. Transfer the sample to a sample container at the sample site. Make the transfer with a ladle rather than pouring to eliminate settling during the pouring process.

1.2. Sludge Discharges into an Open Channel

1.2.1. Sample by passing a sample container through the discharge stream. The sample container opening should be large enough to catch the whole stream rather than just catching the edge or center of the stream.

1.2.2. If access with a sample container is not possible, use a pail or beaker with an extension arm as an intermediate vessel. Transfer the sample immediately to the sample container.

1.2.3. Securely cap sample container and label.

1.3. Sampling Biosolids in Tanks

1.3.1. The purpose of the sampling is to determine the characteristics of the entire mass of sewage sludge.

1.3.2. The entire tank must be well mixed or subsamples must be collected and composited. Take care since tanks might appear to be well mixed but gradients can form. If possible, collect the sample while the tank is being mixed.

1.3.3. Sampling Enclosed Tanks

1.3.3.1. Sample through pipelines entering the digester.

1.3.3.2. Take samples through a minimum of three taps dispersed on the sidewall of the tank. The sample pipes must extend away from the inside walls of the tank.

1.3.3.3. Thoroughly flush the sample line before collecting the sample.

1.3.3.4. Back flush the sample line with tap water after collecting the sample to

avoid the accumulation of sludge and microbial growth as a precaution for the next sampling event at that station.

1.3.4. Sampling Open Tanks

1.3.4.1. Vacuum Flask: Sample by drawing a vacuum on a vacuum-filtering flask by connecting a rigid tube to the filter flask and lowering to the desired depth of the tank.

1.3.4.2. Weighted Bottle: Sample by lowering a weighted sample bottle that can be opened at desired depths.

2. **THICK SLUDGE IN LAGOONS**: Sludges that are above 7% solids start to demonstrate resistance to flow. Sludges in lagoons may thicken to up to 15% solids.

2.1. Thief Sampler: Collect a core sample to the bottom of the sludge layer. The sample obtained with this type of sampler will exclude the overlying water layer that would be included when core samplers are used.

2.2. Weighted Bottle Sampler: Another alternative for collecting thick sludges is to use a weighted bottle sampler that can be opened at desired depths. Where gradients exist, obtain equal amounts of sample from each gradient for compositing.

3. **DEWATERING EQUIPMENT SLUDGE**

3.1. Conveyor Belts: Collect the samples directly from the conveyor belts using a spoon, scoop or shovel.

3.2. Bulk Containers and Piles

3.2.1. Auger with Deeply Threaded Screws: Obtain a cross-sectional sample by turning the auger into the pile and pulling it straight out. Remove the sample from the auger with a spatula, scoop or spoon.

3.2.2. Shovel: Collect subsamples from the pile interior. Sample the pile in proportion to its mass; collecting more samples where the pile is deeper.

4. **DRY SLUDGE**

4.1. Conveyor Belts: Sample when the sludge is being transferred on the sampling belt. Using a spoon, scoop or shovel collect material across the entire width of the conveyor. If it is not possible to collect across the entire width of the conveyor at once, collect samples from various points across the conveyor to represent the entire width.

4.2. Sludge Piles

4.2.1. If the sludge is homogeneous and the sample can be collected within 24 hours of the pile creation follow the sampling procedures in 3.2 of this section.

4.2.2. If the sludge is heterogeneous in nature or sample collection occurs after 24 hours of the pile creation use the following sample procedures:

4.2.2.1. As early as 24 hours after the creation of the pile significant changes may occur that will cause the surface and the interior of the pile to vary significantly.

4.2.2.2. When the piles are more than one day old collect samples from a pile cross-section. Use augers or sample thieves only where there are no large pieces of material or fine composted particles.

4.2.2.3. If augers or sample thieves cannot be used, collect a cross-section sample of the pile with a shovel.

## **FS 2443. Sample Compositing and Size Reduction**

See FS 2420 for a general discussion of sample types. The permit or work plan will specify if composite samples are to be collected and under what conditions. Sample size reduction is often needed when collecting biosolid samples since the individual aliquots collected are usually large in size. The nature of the sample may also make it necessary to reduce particle sizes in order to obtain a representative sample.

### **1. SAMPLE COMPOSITING AND SIZE REDUCTION**

1.1. Do not use automatic composite samplers for the collection of biosolids because of the solids content and viscosity of the sludges.

1.1.1. Automatic samplers, which use pumps to draw samples up a suction tube, will cause solids separation if the flow velocity in the suction and discharge tubes is too low.

1.1.2. The tubing and pump structure will become fouled contaminating subsequent aliquots.

1.1.3. Tubing blockages will occur frequently.

1.2. Use manual composite sampling.

1.3. Compositing and Size Reduction Methods: Collect sample aliquots in individual bottles and composite at the site or back in the laboratory.

#### **1.3.1. Freely Flowing Sludges**

1.3.1.1. Accomplish compositing of smaller samples of freely flowing liquids by pouring them into a larger container with adequate headspace and shaking the sample thoroughly or stirring with a sterile paddle.

1.3.1.2. Pouring off the contents of a larger container into a smaller container is not acceptable. Using a pipet with a wide bore is acceptable as long as the sludge is stirred and the pipet does not restrict any of the particles. Draw the sample into the pipet slowly and move the tip through the sample to minimize selective collection of liquid over solid particles.

1.3.2. Thick Sludges: Compositing and sample size reduction for thick sludges is difficult because shaking cannot mix them.

1.3.2.1. Collect subsamples with a spoon, scoop or spatula for compositing.

- Hand mix each individual sample. Stirring with a mechanical mixer or paddle is often inadequate.
- Take equal amounts of each well mixed individual sample and combine to make one composite sample.

#### **1.3.3. Dry Solid Samples**

1.3.3.1. Homogeneous Samples: Mix the sample by shaking the container. Remove subsamples with a spoon, scoop or spatula to make up the composite sample.

#### **1.3.3.2. Heterogeneous Samples**

- If the particles are large and a number of subsamples must be combined into one composite sample, then shredders, choppers

or grinders may be needed to obtain a representative sample. The laboratory must conduct this activity.

- It is important that the lab and samplers communicate effectively regarding the handling of the samples since particle size reduction is not appropriate if large pieces in the sample are not sewage sludge but materials added for processing the sludge.

## 2. COMPOSITE DURATION

2.1. For bacteriological, vector attraction or viral analyses the maximum time for compositing is one hour. A greater duration may allow for microbiological changes in the first sample aliquot.

2.2. Composite sampling over 24 hours may be done for *Helminth* ova as long as the samples are not exposed to chemical or thermal stress (temperatures not to exceed 40°C, or exposed to such chemicals as ammonia, hydroxides or oxidants).

## **FS 2450. SAMPLING FOR CRYPTOSPORIDIUM AND GIARDIA**

Samples collected for the analysis of Cryptosporidium and Giardia in wastewater are to follow the procedures in EPA Method 1623. In order to meet reporting criteria for certain projects the required sample volumes may need to be modified, where possible, to meet the project data quality objectives.

1. Samples are collected in 10 L plastic carboys or other containers large enough to meet the project data quality objectives.
2. Follow restrictions concerning bacteriological sampling in FS 2005.

## **FS 2460. DOCUMENTATION**

1. Immediately following sample collection, identify each sample container with a unique field identification code.
2. Record sampling information specific to the site while still at the site and meet all of the requirements specified in FD 1000.

## **FS 2470. SAMPLE TRANSPORT AND HANDLING**

1. For information on sample preservation and holding time requirements see FS 1000, Tables FS 1000-4 and FS 1000-5. See the exceptions for the biosolids samples in section 4 below.

2. FS 1000, Table FS 1000-4 includes allowances for automatic samplers. In addition to the requirement of keeping samples cooled with ice or refrigerated during the sampling event (where cooling is applicable to the analytes of interest), there are several considerations to be presented for chemical preservation:

2.1. If discrete aliquots are collected into separate bottles, you may prepreserve the containers with the appropriate chemical preservative or preserve the samples immediately after sampling has been completed.

2.2. If a large compositing jug is used, preserve samples immediately after sampling has been completed.

3. If samples are collected with a composite sampler, the only analyte of interest is Total Phosphorus and the project is unrelated to an NPDES permit, then chemically preserve the sample with H<sub>2</sub>SO<sub>4</sub> (it need not be cooled to 4°C with wet ice). The acid must be in the container prior to sample collection.

4. BIOSOLIDS SAMPLES (maximum holding times, preservation and container type): The holding times, preservation and container type are the same as those listed in Table FS 1000-4 except for fecal coliform.

4.1. Fecal Coliform

Maximum Holding Time	Preservation	Container Type
24 hours	Cool 4°C	Plastic or Glass

4.2. Analytes not in Section 1000, Table FS 1000-4 or Table FS 1000-5.

Analyte	Max. Holding Time	Preservation	Container Type
<i>Salmonella</i>	24 hours	< 10°C	Plastic or Glass
Enteric Viruses	2 hours	Up to 25°C	Plastic or Glass
Enteric Viruses	48 hours	2 to 10°C	Plastic or Glass
Specific Oxygen Uptake Rate	As Soon As Possible	None	Plastic or Glass
<i>Helminth</i> Ova	24 Hours	< 4°C (do not freeze)	Plastic or Glass

5. CRYPTOSPORIDIUM AND GIARDIA SAMPLES

5.1. Ship samples to the laboratory the day they are collected.

5.2. Samples must arrive at the laboratory within 24 hours of sample collection.

5.3. Maintain samples at 0 – 8°C between collection and the time of filtration at the laboratory. Do not allow the samples to freeze.

5.4. Transporting Infectious Materials: The U.S. Department of Transportation (DOT) regulations (49 CFR 172) prohibit interstate shipment of more than 4 L of solution known to contain infectious materials. State regulations may contain similar regulations for intrastate commerce. This method requires a minimum sample volume of 10 L. Unless the sample is known or suspected to contain Cryptosporidium, Giardia, or other infectious agents (e.g., during an outbreak), samples should be shipped as noninfectious and should not be marked as infectious. If a sample is known or suspected to be infectious, and the sample must be shipped to a laboratory by a transportation means affected by DOT or state regulations, it is recommended that the sample be filtered in the field, and that the filter be shipped to the laboratory to avoid violating transport regulations.

**FS 2480. REFERENCES**

1. Florida Department of Environmental Protection, Chapter 62-640, Florida Administrative Code, Effective March 30, 1998.
2. U.S. Environmental Protection Agency, Region 4, Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, May 1996 with 1997 revisions.

3. U.S. Environmental Protection Agency, Office of Water, Part 503 Implementation Guidance, EPA-833-R-95-001, October 1995.
4. U.S. Environmental Protection Agency, Office of Research and Development, Control of Pathogens and Vector Attraction in Sewage Sludge, EPA/625/R-92-013, Revised October 1999.
5. U.S. Environmental Protection Agency, Permits Division, August 1989.
6. U.S. Environmental Protection Agency, Office of Water, EPA 1623, *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA, EPA-821-R-99-006, April 1999.
7. Code of Federal Regulations, Chapter 49 part 172, October 1, 2000.

**APPENDIX FS 2400**  
**Tables, Figures and Forms**

Table FS 2400-1. Wastewater Sampling Equipment

**Table FS 2400-1 Wastewater Sampling Equipment**

<b>EQUIPMENT</b>	<b>USE</b>
Unpreserved sample container Pond Sampler Scoops Beakers Buckets Dippers Other container types	Surface sampling using an intermediate vessel
Peristaltic pump and tubing Sample container	Surface sampling directly into, or pumped into, sample container
Kemmerer Van Dorn Nansen Alpha Bottle Beta Bottle Niskin Equivalent devices Double check-valve bailer Pole and attached container (removable top) Peristaltic pump and tubing	Specific-depth grab sampling
Automatic composite samplers	Composite sampling
Sample Containers Buckets Scoops Dippers Beakers Vacuum flask Weighted bottle sampler	Free flowing sludges
Thief sampler Weighted bottle sampler	Thick sludges
Spoon Scoop Shovel Auger	Sludges from dewatering equipment
Auger Shovel Sample thief	Dry sludges